

Sputtered Silver Films To Improve Chromium Carbide Based Solid Lubricant Coatings for Use to 900 °C

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LUBRICANT COATINGS FOR USE TO 900 °C (NASA)
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SPUTTERED SILVER FILMS TO IMPROVE CHROMIUM CARBIDE BASED SOLID
LUBRICANT COATINGS FOR USE TO 900 °C

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SUMMARY

Thin silver films, 250 to 3500 Å thick, were sputtered onto PS200, a plasma sprayed, chromium carbide based solid lubricant coating, to reduce run-in wear and improve tribological properties. The coating contains bonded chromium carbide as the wear resistant "base stock" with silver and barium fluoride/calcium fluoride eutectic added as low and high temperature lubricants respectively. Potential applications for the PS200 coating are cylinder wall/piston ring lubrication for Stirling engines and foil bearing journal lubrication.

In this preliminary program, the silver film overlay thickness was optimized based upon tests using a pin-on-disk tribometer. With this apparatus the PS200 coating is applied to the disk and slid against a clean metal pin. The friction and wear studies were performed in a helium atmosphere at temperatures from 25 to 760 °C with a sliding velocity of 2.7 m/s under a 4.9 N load.

At room temperature there is a direct relationship between the silver film thickness and the ratio of pin wear to coating wear. The silver film overlay reduces counterface wear up to a thickness of about 1500 Å. Thicker films cause plowing of the silver by the counterface material and do not further reduce wear. Since silver is primarily a low temperature lubricant, no functional relationship between the silver overlay thickness and wear was observed at 350 and 760 °C. However, the data indicates that the silver overlay reduces the initial abrasiveness of the coating and significantly reduces run-in wear.

Films between 1000 and 1500 Å provide the best lubrication of the counterface material. The films enrich the sliding surface with lubricant and reduce the initial abrasivity of the as ground plasma-sprayed coating surface.

INTRODUCTION

The lubrication of sliding contacts at high temperatures has been an area of increasing interest due to the need for high temperature lubrication in advanced heat engine applications (e.g., Stirling engine, Low Heat Rejection diesel, advanced turbomachinery, etc. (ref. 1)). To help satisfy these lubrication requirements a plasma-sprayed, chromium carbide based, self-lubricating composite coating has been developed at NASA, Lewis Research Center (ref. 2).

The base material of this coating system is metal-bonded chromium carbide. It is a good candidate for high temperature wear applications because of its thermal and chemical stability and excellent wear resistance. However, when used alone as a coating for sliding contacts chromium carbide exhibits poor

friction characteristics. By adding the solid lubricants silver and barium fluoride/calcium fluoride eutectic, friction and wear properties of the Cr_3C_2 base coating can be significantly improved (ref. 3). Silver, because of its low shear strength, is a good thin film lubricant at low temperatures and the eutectic, which undergoes brittle to ductile transition around 500 °C, is a good high temperature lubricant (ref. 4). Thus, the coating is designed to lubricant over a wide temperature range from room temperature to over 900 °C.

Previous tests with this coating system indicated that initial counterface wear rates are significantly higher than steady-state wear rates (ref. 2). Depending upon the application, run-in wear can greatly reduce the overall useful life of a component. For this reason, it is desirable to reduce the run-in wear especially of the counterface material which frequently has lower allowable volumetric wear than the coating that they slide against (e.g., piston rings compared to cylinder wall coatings).

Preliminary unpublished work with the PS200 coating indicated that run-in wear, especially of the counterface material, can be reduced by applying a thin film of silver over the plasma-sprayed coating prior to sliding. The silver provides a soft lubricant film between the counterface material and the initially rough (surface finish $>1 \mu\text{m rms}$) coating which reduces wear of the counterface material. These tests indicated that the tribological performance of the silver coated PS200 coating is significantly affected by the silver film thickness. References 5 and 6 are general references which describe the use of thin soft metallic films to reduce friction and wear.

This paper describes a program to further study the effects of silver overlays on the PS200 coating. Using a pin-on-disk tribometer, a preliminary optimization of the film thickness was performed to find a range of thicknesses which demonstrate improved tribological properties. The tests were run in helium gas to provide an inert atmosphere which simulates conditions that may be encountered in a Stirling engine.

EXPERIMENTAL MATERIALS

Wear Pins

The wear pins are made of a hardened cobalt-chromium based alloy. It is chosen because of its strength at elevated temperatures, chemical and thermal stability, and suitability for use as an engine component material. This alloy has also given consistent, repeatable friction and wear results when slid against the PS200 coating (ref. 2). The test pins are hemispherically tipped with a radius of 4.76mm and are 20 mm long. The nominal composition of the alloy by weight percent is: 59 Co, 30 Cr, 4 W, 2 Ni and 5 other. The Rockwell Hardness of the pins is 40 C and the surface finish is $0.15 \mu\text{m rms}$.

Test Disk Substrate

The test disks are 63.5 mm in diameter, 12.7 mm thick and made of a high temperature precipitation hardened nickel chromium alloy hardened to Rockwell 40 C. The disk composition by weight percent is: 70 Ni, 15 Cr, 7 Fe, 2.5 Ti, 1 Al, 1 Mn, 1 Co and 2.5 other.

Disk Coating: Materials and Preparation

PS200 coating used in this study is a plasma sprayed coating prepared from powders that contain 80 wt % metal-bonded Cr_3C_2 , 10 wt % silver and 10 wt % $\text{BaF}_2/\text{CaF}_2$. This coating is chosen because it gives repeatable results when slid against the chosen pin material and because a relatively large data base exists for it.

Three components are used in the PS200 plasma sprayed coating. The silver metal and the metal-bonded Cr_3C_2 are available as plasma-spray powders. The $\text{BaF}_2/\text{CaF}_2$ eutectic powder was made by prefusing and regrinding the individual fluorides prior to mixing with the other coating components. The following procedure was used to prepare the eutectic powder. BaF_2 and CaF_2 reagent grade purity powders (purity >99.9 percent) were blended in eutectic proportions and placed into a clean nickel crucible. The powders were fired in a nitrogen atmosphere tube furnace at 1100 °C until completely fused. The resulting melt is allowed to cool to room temperature in nitrogen. It was then crushed in an opposing plate type mechanical crusher into 1 to 2 mm diameter pieces. These were further ground to a fine powder in a ball mill with Al_2O_3 crushing stones. The ground eutectic powder was then sieved to obtain the correct size powder for plasma spraying. X-ray diffraction confirms that eutectic powder prepared in this manner is free from significant contaminants. The three powder components were blended together prior to plasma spraying onto the test disks. Table I gives the mesh size and exact composition of the coating components.

The disks were first sandblasted then, a thin bond coat (0.076 mm) of Nichrome (80 wt % Ni, 20 wt % Cr) powder was plasma sprayed onto the roughened surface. The PS200 powder mixture was then plasma sprayed over the bond coat to a thickness of 0.38 mm. The plasma spray parameters are given in Table II. Wet chemical and spectrographic analysis of the resulting coating indicated that its composition is approximately the same as the PS200 powder prior to plasma spraying.

The coating was diamond ground to give a total coating thickness (bond coat plus lubricant coat) of 0.25 mm. Appendix A describes the grinding procedure. Figure 2 is an illustration of hypothetical ground surfaces. Figure 3 is a photograph of a diamond ground part. During grinding particular care must be taken to prevent smearing of the coating and to prevent removal of the soft phases, namely the silver and the eutectic. These problems can be avoided by employing the recommended depths of cut with a well dressed diamond wheel. Lapping procedures should be especially avoided because of their tendency to preferentially remove the soft lubricant phases from the coating surface.

Silver Film Deposition

Prior to silver coating the ground plasma sprayed PS200 coatings, the specimens were heated in a vacuum oven at 150 °C for 3 hr to remove any volatile residue remaining from the grinding operation and subsequent handling. The specimens were cleaned with pure ethyl alcohol and lightly scrubbed with levigated alumina then rinsed with deionized water and dried.

The PS2000 disk specimens were coated with silver films from 250 to 3500 Å thick using a magnetron sputter deposition system with argon as the ionization gas. The sputtering parameters were; power: 0.05 kW, argon flowrate: ≈ 10 cc/min, chamber pressure: 50 mtorr, and target to specimen distance: ≈ 25 mm. The silver coating thickness was calculated by multiplying the deposition time by the deposition rate. The deposition rate was determined from a series of calibration trials in which varied thicknesses of silver were sputtered onto polished test disks and measured using a computer controlled profilometer. The slope of the plot of coating thickness versus time is the deposition rate. For these experiments, the rate is 150 Å/min. Following this procedure, the thickness is known to within ≈ 50 Å.

APPARATUS AND TRIBOTEST PROCEDURE

Apparatus

A pin-on-disk apparatus was used in this study (fig. 1). With this apparatus, a hemispherically tipped pin is loaded against a rotating disk by means of dead weights. The normal load was 4.9 N. The friction force was continuously measured with a temperature compensated strain gage bridge. The pin wears a 51 mm diameter wear track on the disk. The disk wears a flat circular wear scar on the pin. Sliding was unidirectional and the velocity in these experiments was 2.7 m/s. The disk was heated by a low frequency induction coil. Disk surface temperatures were monitored on the wear track 90° ahead of the sliding contact with an infrared pyrometer capable of measuring temperatures from 100 to 1400 °C with ± 5 percent accuracy.

Procedures

The test duration was 3 hr, 1 hr at each of the three test temperatures in the following order, 760, 350, and 25 °C. If the specimens were run first at 25 °C, then at 350 °C and then at 760 °C, the results at 25 °C, would not agree with data collected during subsequent room temperature tests after the specimens had been run at elevated temperatures. This is due, in part, to diffusion of the silver overlay into the PS200 coating, the majority of which occurs during the first few minutes of heating to elevated temperatures. By testing the specimens first at 760 °C then at 350 °C and finally at 25 °C, however, the data taken at 25 °C agree well with steady state values.

Rider wear was measured every 20 min by removing the pin and measuring the resulting circular wear scar on the hemispherical surface from which the wear volume was calculated. Locating dowels on the rider holder assured accurate relocation of the pin. Disk wear was measured after each hour by recording a surface profile across the disk wear track, computing the area of removed/displaced coating and multiplying by the circumference of the wear track to obtain the wear volume.

Prior to testing, the test chamber was closed and then purged for 10 min with nitrogen gas. The helium was then purged for 10 min before the test was begun. After elevated temperature tests, the specimens were cooled to below 150 °C before opening the test chamber to inhibit specimen oxidation which might have invalidated later analysis.

EXPERIMENTAL RESULTS

The test results are given in Table III. Depending on the thickness of the silver film overlay, the friction coefficients ranged from 0.16 ± 0.02 to 0.34 ± 0.04 at room temperature, 0.16 ± 0.02 to 0.28 ± 0.03 at 350°C , and 0.20 ± 0.03 to 0.40 ± 0.04 at 760°C . Wear factors ranged from $(8.0 \pm 1.5) \times 10^{-8}$ to $(1.0 \pm 0.2) \times 10^{-5} \text{ mm}^3/\text{N-m}$ for the pin and $(1.8 \pm 0.3) \times 10^{-6}$ to $(2.3 \pm 0.4) \times 10^{-5} \text{ mm}^3/\text{N-m}$ for the coating. An explanation of the wear factor, k , and its derivation are given in appendix B.

The tribological performance of the PS200 coating without a silver overlay, the control case, was good. Friction coefficients were between 0.19 ± 0.02 and 0.26 ± 0.03 and average wear factors were reasonably low. When a 1000 \AA thick silver overlay was applied to the PS200 coating the test results were significantly improved. Friction coefficients were between 0.16 and 0.20 and total wear of the pin material was reduced by a factor of three.

Discussion of Experimental Results

The initial purpose of this program was to reduce the run-in wear of counterface materials when slid against the PS200 coating. By achieving this goal, the overall life of a device or an application can be greatly increased.

Figure 4 is a plot of pin wear volume versus sliding distance for both the control case and the PS200 coating with a 1000 \AA silver film overlay. Though the graph incorporates data from three different test temperatures, the curve is relatively smooth. The data indicate that the total pin wear after 3 hr of sliding is about three times greater for the control case than for the pin slid against the PS200 coating with a 1000 \AA silver overlay film. Considering experimental error, the slopes of the curves ($(0.12 \pm 0.01) \times 10^{-2} \text{ mm}^3/\text{min}$ for the control coating and $(0.10 \pm 0.01) \times 10^{-2} \text{ mm}^3/\text{min}$ for the 1000 \AA silver overlay case) are approximately the same after 100 min of sliding. Thus, the silver film did not significantly affect the steady state wear properties of the PS200 coating. Rather, the data suggests that the difference in total pin wear is due to the reduction in the run-in wear by the use of the silver overlay. Similar results were obtained for film thicknesses of 750 , 1250 and 1500 \AA . Thinner films did not seem to significantly reduce run in wear and plowing of the silver film was observed when thicker films are tested. This plowing lead to excessive silver transfer to the pin and higher friction especially at elevated temperatures. Thus, from these preliminary tests, a silver film overlay thickness between 750 and 1500 \AA provided the greatest benefit.

When investigating the abrasivity of one material to another it is instructive to consider the ratio of the wear factors (i.e., the wear factor of the pin divided by the wear factor of the disk coating). The larger the ratio, the more abrasive the coating is to the pin and vice-versa.

Table IV contains the wear factor ratio tabulated with the silver film thickness and test temperature. Figures 5(a) to (c) are plots of the wear factor ratio versus film thickness for the three test temperatures. When these plots are inspected, several trends become apparent.

At 25°C the ratio is about 0.10 for the control case (with no silver overlay). As the silver film thickness is increased from 250 to 1500 \AA , the ratio

is decreased from about 0.11 to 0.02. That is, the silver film reduces the abrasivity of the coating to the pin up to a film thickness of 1500 Å. Films thicker than 1500 Å do not result in a further wear factor ratio reduction.

At 350 °C there is no clear trend in the data. This may be due, in part, to the fact that at elevated temperatures, silver has low compressive strength and, by itself, is not an adequate lubricant.

At 760 °C there is no relationship between the film thickness and the ratio, but the silver films did reduce the ratio approximately a factor of two compared to the control case. One plausible reason for this behavior is that the tests at 760 °C were the initial tests for each specimen. In these cases, the silver film merely served to smooth out and fill in the imperfections in the plasma-sprayed surface leading to reduced pin wear. But since the silver has low compressive strength at 760 °C the benefit was not a function of silver film thickness.

The application of silver films around 1000 Å thick reduced the run in wear of the pin without causing plowing and excessive silver transfer to the pin surface. The silver film acted as a run-in lubricant which smoothed out the surface and reduced the initial abrasivity of the PS200 coating to the counterface material.

CONCLUSIONS

The following conclusions may be drawn from this preliminary study:

1. The run-in wear of counterface materials, when slid against PS200, appears to be reduced by applying thin silver films over the as ground, plasma-sprayed surface.
2. The friction and wear of the counterface material is affected by the thickness of the silver film overlay. Films between 750 and 1500 Å thick seem to improve the counterface wear properties without adversely affecting the friction coefficient or coating wear.
3. the reduction in counterface wear obtained by this method may significantly increase the overall life of a component or application.

APPENDIX A

RECOMMENDED GRINDING PROCEDURE

1. Use diamond grinding only.
2. Use water as lubricant-use no oil.
3. Initial grinding depth should be 0.0250 mm.
4. Final cuts should be 0.010 to 0.0150 mm.
 - Taking too deep a cut, i.e., 0.10 mm, will pluck softer phases (Ag and $\text{BaF}_2/\text{CaF}_2$) from surface.
 - Taking too light a cut, i.e., less than 0.010 mm, will smear the metal-bonded chromium carbide. This will result in an "orange peel" type finish.
5. Ground surface should be matte not glossy and have a speckled appearance representing the three separate phases.

APPENDIX B

EXPLANATION OF WEAR FACTORS

The wear factor (K) used in this paper is a coefficient which relates the volume of material worn from a surface to the distance slid and the normal load at the contact. Mathematically, K is defined as:

$$K = \frac{V}{(S \times W)}$$

where

W the normal load at the sliding contact in N

S the total distance slid in m

V the volume of material worn away in mm³

The physical interpretation of the numeric value of the K factor is as follows:

K 10⁻⁴ mm³/N-m high wear

K 10⁻⁵ to 10⁻⁶ mm³/N-m moderate to low wear

K 10⁻⁷ mm³/N-m very low wear

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TABLE I. - COMPOSITION OF THE THREE
MAJOR COATING COMPONENTS

Component	Composition, wt %	Particle size
Bonded chromium carbide		
Ni	28	-200 + 400 Mesh
Al	2	
Cr ₃ C ₂	58	
Co	12	
Silver metal		
Ag	100	-100 + 325
Prefused eutectic		
BaF ₂	62	-200 + 325
CaF ₂	38	

TABLE II. - TYPICAL PLASMA SPRAY PARAMETERS

Parameter	Material, value
Arc gas, 1.4 m ³ /hr	Argon
Powder carrier gas, 0.4 m ³ /hr	Argon
Coating powder flow rate, kg/hr	1
Current, A	450 to 475
Voltage, V	32
Gun to specimen distance, mm	~150

TABLE III. - FRICTION AND WEAR TEST RESULTS

[Test conditions: 4.9-N load, 2.7-m/s sliding velocity, Helium atmosphere.]

Test temperature, °C	Silver overlay thickness, Å	Friction coefficient	Pin wear factor		Coating wear factor	
			cm ³ /cm-kg	mm ³ /N-m	cm ³ /cm-kg	mm ³ /N-m
760	0	0.26	1.10x10 ⁻¹⁰	1.12x10 ⁻⁶	8.75x10 ⁻¹⁰	8.93x10 ⁻⁶
	250	.34	.21	.21	5.00	5.10
	500	.29	.84	.86	11.20	11.43
	750	.24	.87	.89	14.60	14.90
	1000	.20	.42	.43	7.01	7.15
	1250	.23	.66	.67	9.30	9.49
	1500	.31	.48	.49	14.00	14.29
	2000	.33	.98	1.00	22.00	22.45
	3500	.40	.73	.74	20.00	20.41
350	0	0.19	0.40	0.41	2.80	2.86
	250	.28	.69	.70	1.20	1.22
	500	.24	.51	.52	4.80	4.90
	750	.19	.23	.23	3.60	3.67
	1000	.16	.16	.16	2.96	2.96
	1250	.19	.33	.34	1.02	1.02
	1500	.19	.40	.41	4.08	4.08
	2000	.22	.17	.17	4.08	4.08
	3500	.21	.56	.57	3.37	3.37
25	0	0.19	0.18	0.18	1.70	1.73
	250	.34	.32	.33	3.10	3.16
	500	.25	.49	.50	5.60	5.71
	750	.22	.29	.30	4.40	4.49
	1000	.17	.08	.08	4.10	4.18
	1250	.20	.08	.08	1.65	1.68
	1500	.21	.20	.20	4.70	4.80
	2000	.16	.06	.06	1.00	1.02
	3500	.20	.13	.03	1.70	1.73

TABLE IV. - WEAR FACTOR RATIO

Test temperature, °C	Silver film thickness, Å	K _{pin} /K _{coating} ratio
760	0	0.120
	250	.042
	500	.075
	750	.059
	1000	.059
	1250	.070
	1500	.034
	2000	.044
	3500	.036
350	0	0.142
	250	.058
	500	.106
	750	.063
	1000	.055
	1250	.330
	1500	.097
	2000	.042
	3500	.169
25	0	0.105
	250	.106
	500	.087
	750	.075
	1000	.019
	1250	.050
	1500	.042
	2000	.062
	3500	.076

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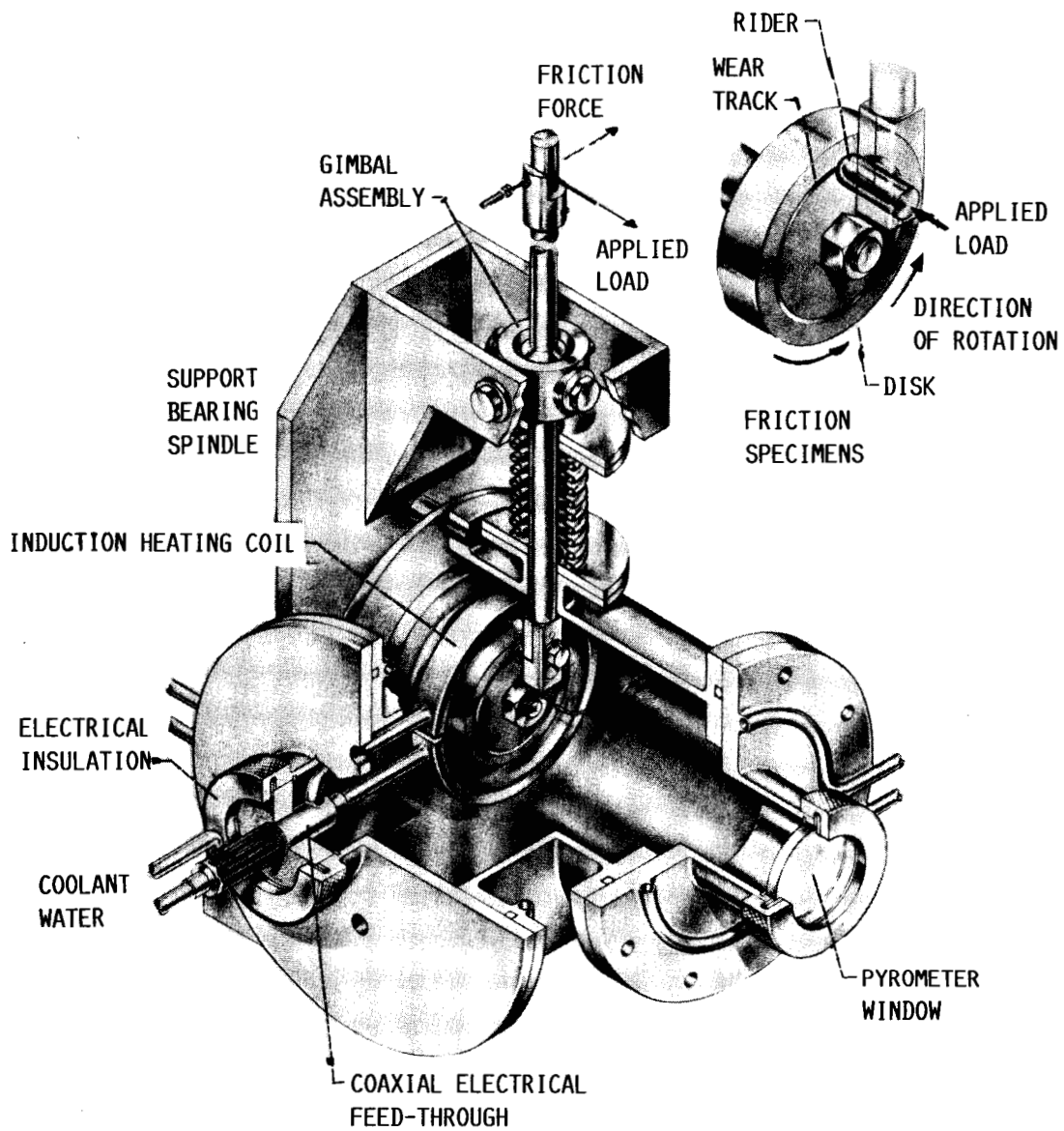
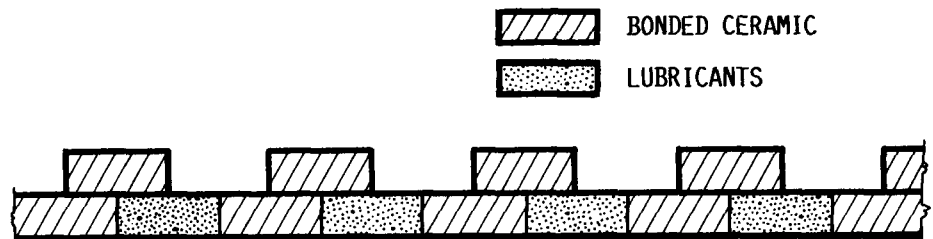


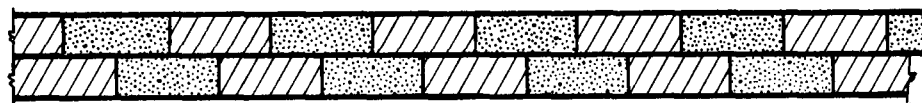
FIGURE 1. - HIGH-TEMPERATURE FRICTION APPARATUS.



GROUND COATING FROM WHICH SOFT LUBRICANTS HAVE BEEN "PLUCKED" FROM THE SURFACE. POOR SURFACE.



GROUND COATING IN WHICH NICKEL BOND MATERIAL HAS BEEN SMEARED OVER COVERING LUBRICANTS. POOR SURFACE.



CORRECTLY GROUND COATING. BOTH THE LUBRICANTS AND BONDED CERAMIC ARE PRESENT AT SURFACE.

FIGURE 2. - HYPOTHETICAL CROSS-SECTION ILLUSTRATION OF DIAMOND GROUND SURFACES.

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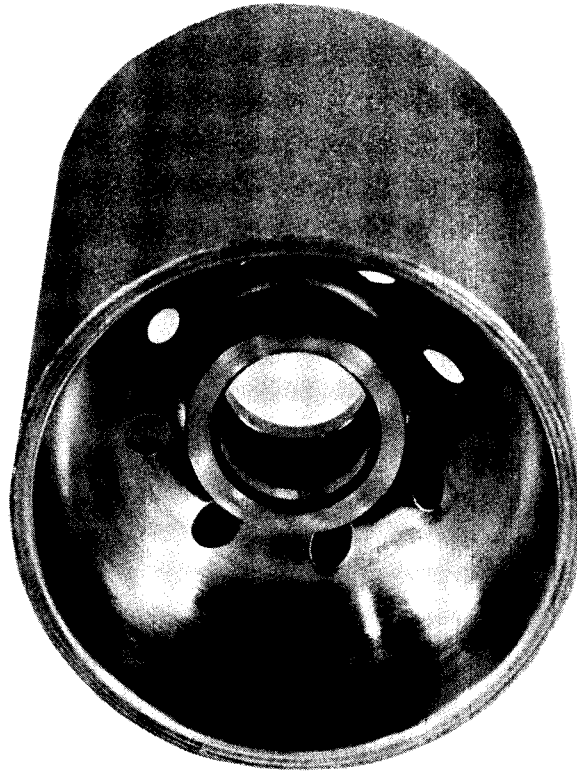


FIGURE 3. - DIAMOND GROUND PART.

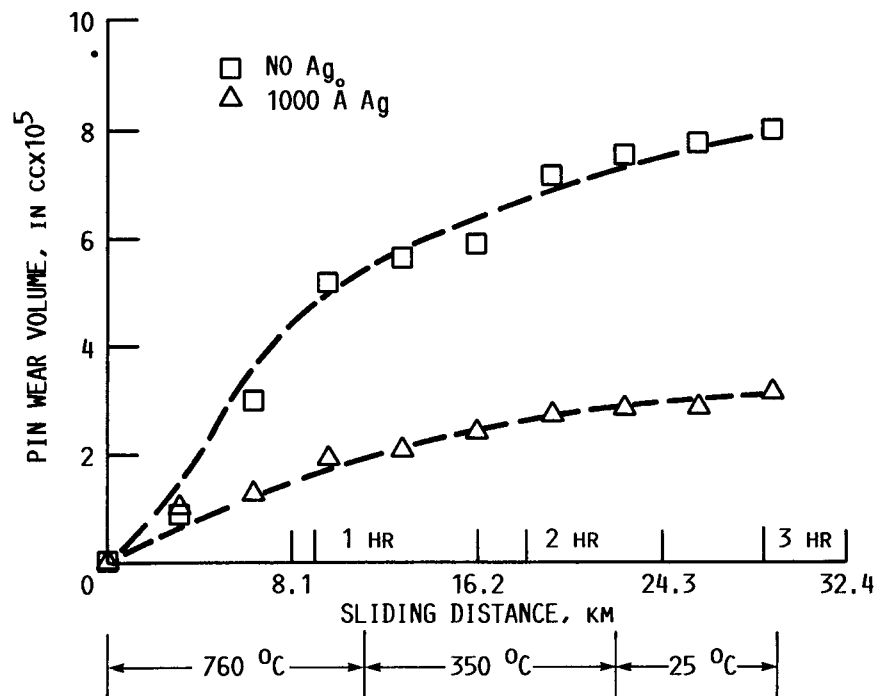


FIGURE 4. - PIN WEAR VOLUME VERSUS SLIDING DISTANCE.
TEST CONDITIONS: 4.9 N LOAD, 2.7 M/SEC SLIDING VE-
LOCITY, HELIUM ATMOSPHERE.

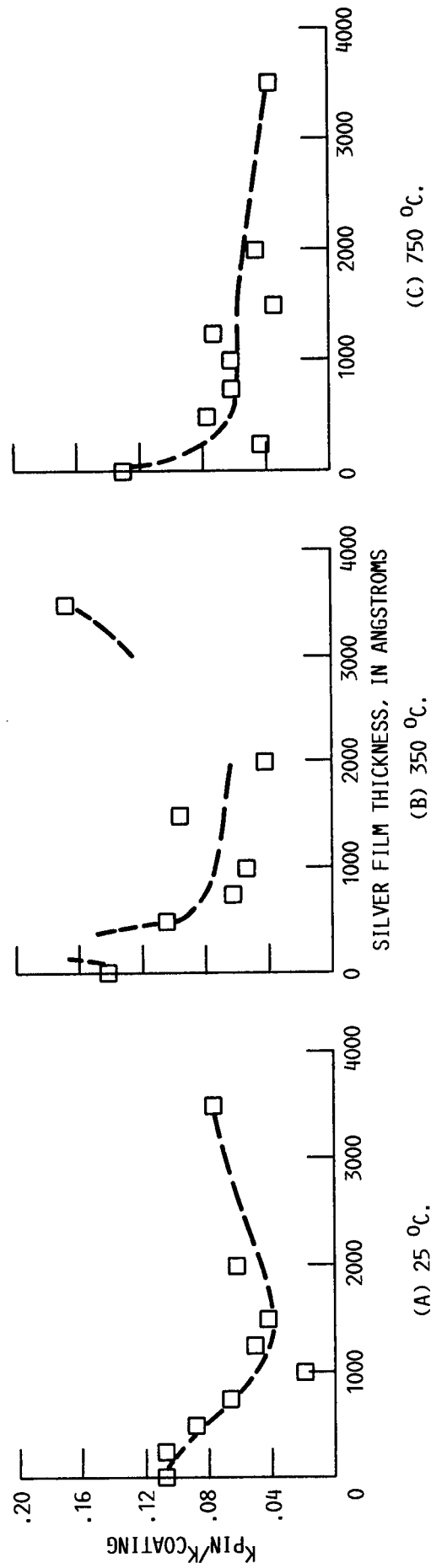


FIGURE 5. - WEAR FACTOR RATIO VERSUS SILVER FILM OVERLAY THICKNESS.



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